Notebook Series -- Notebooks by Edison
Notebook, N-18-12-26.2

This notebook is a continuation of N-18-11-26. It was used by Edison during the period December 1918-February 1919 for notes on processing lithium. Among the ores used for experimentation are lepidolite (lithium mica), amblygonite, and spodumene. The entries describe different fusing, filtering, and leaching steps, along with the reagents employed, and note the presence of a lithium "line" as indicated by a spectroscope. Also included is a list of organic solutions in which Edison tested the solubility of lithium chloride, along with notes of results from the tests described in N-18-12-21 [not selected]. The notes indicate that Ludwig F. (Louis) Ott assisted in some of these experiments and performed assays on some of the final residues. The front cover is labeled "Lithia Dec 26 1918." The pages are unnumbered. Approximately 100 pages have been used.

I notice on Dec 26 – the Cold Dup gives a very bright Li line,

on 19th 4.20 pm

No 7 Ambligonite –

Shows that, when Amblig is quenched, reground, ignited again & reground fine – that in 4 days on hot plate on a Butler dish & followed that most of Lithia comes out. If now – the residue w/ Ephemeral + fresh water put in with 10 cc HCl more & kept for 3 days the residue is small & practically all the Lithia is out – testing dry residue in flame gives poor line

Think that 7 cc HCl 1st Ephemeral & 4 on 2nd will be commercial if finely ground

C13

Chunk of Pink Lepodolite ignited quenched - powdered put in Casserole Dec 19th 9 am - 10 grms with 3 cc Conc HCl at night put in 5 cc HCl more. Total 8 cc HCl.

Taking off hot plate 27th Dec 930 am it was nearly dry & hard Caked together, added about 100 cc water & broke up Caking but still sandy after boiling 1/2 hour -

It was only faintly acid so I added a little HCl - filtered

Lime process –

After acting on ore for several days boil the solution with ore & all in & then add proper amount of W Va lime 3/16 size & stir & keep boiling for a little while – Then filter hot, filters rapid & good, then wash several times with minimum of hot water till Lithia line disappears in direct vision spectroscope, Boil the clear solution & then add Oxalic acid till its on the verge of being acid this throws down all the Lime, filter hot, filters rapid –
The liquid will have only the Chlorides,

13 -

Ambliqonite - Chunk ignited & quenched this is dried and ground up to about 100 mesh & ignited in a mond box to a temp where its semi melted & sticks together quench in cold water, Then powder up roughly & assay samples are ground to 200 mesh -

This is 10 grams of Ambliqonite 10cc 37% HCl, diluted with 40cc water.

On butter dish over hot plate Dec 27 5.20 pm

The temp of the solution well over 75° Cent. - probably less as butter dish is old thick one

Final Test of Solubility of Lithium Chloride in Organic Solvents
Solutions filtered

Amyl Acetate	good
Amyl Alcohol	good
Acetone	good
Pyridin	good
Benzaldehyde	weak
Dichlorohydrin	fair
Piccolin	good
Ortho Monochlorophenol	fair
Methylene chloride	good
Butylic Alcohol	V good
Guaicol	Try again

Amyloxide butyric	good
Aether Butyric	V good
Metaldehyde.	good
Orthomonochloraphenol.	faint
Trioxsol ?	fair
Ergosaure Anhyd ?	good
Phenylhydozine —	good

14 –

10 grms Ambligonite treated as to equilibrium like # 13.

40 cc Alcohol
10 cc Conc HCl –

Dec 28 on warm on wood piece – 3 20 pm –

Carb Ammonia dont precip Li_2CO_3 from Chloride di but Carb Soda does —

~~Remarks —~~

~~The solution from No 4 deposited, filtered on (see book 1) in Several days — poured all into a Crystallizer as~~

Continued from old book

Dep. No 4 of old book

After washing the Lime sludge well there was still a faint Lithia Lime + sludge — itself gave brilliant lines — after exhausting all night in Cassenales filtered off + treated with Oxalic to throw down lime — boiled down to few CC + added Carb Ammonia — Thus precipitated Li Carbonic + filtered it off to about 1/5th (was CC weight) of the first precipitation with Carb Soda —

The solution being filtered off from Li_2CO_3 there was

still a brilliant line in wash water & good line even when diluted to 250 cc

I then evaporated it down having added HCl & nitric ac to oxidize the ammonia & get a residue — Evapt'd to dryness of course I then treated dry residue with alcohol & this gave a brilliant line while the residum that didn't dissolve in alcohol was acidulated with HCl & put in test tube 3/4 full water it gave very poor line showing most of the lithia dissolved in the alcohol —

This is a fine way to get last traces of LiCl out of final products (12) alcohol which dont dissolve but traces Na + KCl or other things —

I still have the lime sludge & will acid elute it with HCl & see if I can get more out,

On Dec 28 5.30 pm plenty HCl

I find at 12 noon Dec 28 that the sludge water is entirely too strong in Lithia, and that the original ore was not exhausted of Lithia.

The Original ore should be finer ground and exhausted 2 or 3 times without an amount of acid to be determined, I think there was not acid enough to start that at least 15 cc 37% HCl

No 4

should have been used in the first place, or perhaps 10 cc & then filtered & washed & then 5 cc more put in for couple days — & finally ~~the~~ the whole limed, with unequal ones in or out, this is to be determined —

The 1st Crop of Carbonate Li weighed 1.560 —

Second weighed 0.164

90.5% 1st 9.5% 2nd with more mixed

The question is how pure it is — 2pt shows good line,

1st Jany 1919 –
9.30, filtered off solution about 250 cc cant get any Lithia line This is 2nd leach
Saved residue as no longer giving Li –
11 AM 1st Residue made acid by HCl
1130 am – fairly good Lithia line –

10 30 am Jany 2" dont get any line of Li in 250cc of 2nd filtrate strong with HCl
This is OK

Evap down water extraction solution to 100 cc – ~~[illegible]~~ poured off all but 20 cc – diluted this till 400 cc when line very faint & like 31 mlg Li ~~[illegible]~~ in 250 cc – ~~Equals 1000 cc solution~~ 1/80 of the Li total –
The acid solution had to be diluted to 1000 cc to be as faint as 31 mlg standard according to this water solution
see page beyond

Some (?) mixed

16 = Lepidolite
2 leaches only

Fusion of 25 grms Lepidolite 25 grms Plaster Paris 25 grms Salt, Fuses easily to thick viscous liquid – Kept it fused 20 minutes Top Crucible below red, put top & Crucible in water dissolved all out Evaporated water & mix down to dry and ground fine –

This sample totals 30 grms 10 salt 10 lep 10 plaster paris supposed to leach in water only

On hot plate Dec 31 noon
LOST = 1/4 of water over boiled & slopped in flask because cork in too tight, will run it anyway

only took out 1/2 which is bad. perhaps blunder do something in this as a reason for poor showing as compared to good which has none

Probably wants Lining before Water Exhaustion

The Original water solution in 200 cc Took 20 cc & diluted it to 200 cc then discarded 100 cc & diluted the remaining 100 to 200 cc = 20 times dilution

Lime pest acc line with less than 31 standard

1/10th –

Something Mixed as I record already tested Water Sol

I think I notice that filtrate (2nd) was much stronger when fresh & it may be dri reverts – however its unpalatable

1030 am Jany 2 – scarcely get a Li line with the 250 cc 2nd filtrate cooler ~~[illegible]~~ with complete HCl – This is OK

The water Extraction has 12 times more Li than the final HCl Extraction in fact this seems perfect if the Spod hasn't a lot locked up that will not come out by prescon & acid –

The acid solution after all night diluted to 500 cc just faint Li line, & this lifted I think I would have got out by boiling water –

17

Duplicate of 16 – Except its Spodumene

On hot plate Dec 31 noon
1st wash filtered off 6.30 pm 31st
Jany 1st 9.30 am filtered off solution 2nd wash been in all night, scarcely get Lithia line
Dried Residue, as no longer gives Li by H_2O

2 Leaches only – now evap to dry

11 am 1st Residue made acid by HCl

1130 am weak Lithia line –

As 16 17 + 8 after extraction with water fully – the acid brings out more Lithia, this shows proportions of $CaSO_4$ + salt not quite exact, it wants longer fusion perhaps to act on all the ore also possibly finer grinding –

17 Continued

The final water after Evap to dry was dissolved in minimum water but left a residue ~~that~~ after filtering - the water was more than simple to decation the alkaline -

This water 100 cc was diluted to 1000 cc Line fine

(500 cc set aside)

Remaining 500 cc diluted to 1000 Line good, (500 cc of this) was set aside,

Remaining 500 cc diluted to 1000 cc - (500 of this set) aside, the remainder diluted to 1000. This gave very weak line. Fragile

Deke 31 July Standard

#16

1030 am, get a faint strong line in final washer of 2nd filtrate strong with HCl — well see if some present of di

The acid HCl from residue after thorough water Extraction, diluted to 2000 cc line fair — Took 1/2 away & the remainder 1000 cc & withdrew 500 cc or what is same thing took 1500 cc away or 3/4 Then diluted the other 1/4 500 cc to 1000 which gave line almost like 31 Mg standard in 250 cc.

This seems poor & its probably fluorine or not Enough gypsum to take care of fluorine —

over

18 -

Dup of 16

Except Ambligonite on hot plate Dec 31 noon

1st wash filtered off 5 30 pm -

2nd wash filtered off 9 30 am January first 1919

~~Will get residue back for a 3rd wash as it shows a pink back distinctly~~

Cant get a line in flume after 2 small after washes of residue

2 washes takes practically all out -

11 am 1st Residue made acid by HCl at 1130 strong Li line

Took the water Exhaust Col
200 cc
abstracted 150 cc
50 cc diluted To 200 cc 25%

Abstracted 150 cc
diluted the 50 cc to 200 cc 6 1/4%

abstracted 150 cc
diluted the 50 cc remaining
to 200 cc this gives
line a little darker
than 31 mg 250 cc standard
1.53%

Fused 25 Lep
12 Cluster Pains
12 Salt
12 Lime

It dont fuse to my cons liquid
just fruts — Think that would
do —

Will try 25 Lep
25 Salt
12 Lime

19 = Jany 3rd 1919

25 lepidolite
25 salt
12 quicklime -

Took 25 grams of this mix which gives 10 grams of lepidolite.

Add 10 grams Caustic W Va Lime in addition -

200 cc water - added lime after mix was hot.

When putting in lime boiled over + large amount of mix lost

20 = 1 + 2nd washed put together
evaporated down to 340 cc —
+ diluted by 10 sections of 340 cc
each total 3740 cc
when the line was about
same strength as standard
solution of 62 mlg LiOH in
250 cc water —
15 times weaker
This would give 930 Mlg
Mlg LiOH. —
The solution has HCl put in,
186 lbs LiOH, to Ton —
1/2 hour after HCl in the residue after
2nd Exhaustion, could only get
a very v faint line
Boiling 1/2 hour goes neutral — & add
more HCl & boil again —
With 125 cc scarcely see line
much less than 31 mg standard
LiOH practically all out on 1st wash
all on 2nd, 3 lbs left [illegible] on 2%.

This is OK & does
20 — the business for Lep
mix
25 Lepidolite
25 Salt
37 Plaster Paris —
200 cc water — in beaker

after boiling add gradually
10 grams W/a quick lime
probably be better slake it to mush
& then put in as piece 1/8 @ 1/4 don't go to piece
1st filtration, about 250 cc shows
very brilliant line — not all out
yet by water 1st filtration still going
on —

2nd filtration 250 cc Count 200
line — When Evaporated
down to about 150 cc can
just see it Extremely faint —

fused at yellow with whitish
aspect, about 1 hour to like
Summer butter

No 20 — work for

Lep —

Ton Lep 200 mesh or finer pre previously ignited ??
1½ Tons Plaster P (or semi dehydrated) gypsum
1 Ton Salt,
all mixed and blended till thick pat — fused to yellow white for say 1 @ 1½ hours —
~~[illegible]~~ Now we put in water to get out Crucible and dried the water out,
then pounded it 200 mesh and Leached ~~after~~ adding 1 ton Caustic Lime & boiling

Hence raw materials

1 Ton Lep
½ Plaster P or say 2 Ton Gypsum
Ton Salt
Ton Caustic Lime —

Possibly can reduce these quantities,

10 amblygonite - Muffled & grounded
then powdered & fused
in HCl - with just enough water
to cover over the Lamp bulb
& 1/4" above, for 6 days -
got Li Line detected with
20000 cc as quad or
62 mlg in 250cc standard &
yet all of the solution not filtered
out of ore -
There is something wrong as
this would be 900 lbs in 1
Ton - Its probable the
standard is wrong & not
really deep Li OH.

It needs to be vigorously boiled probably to increase crystallization possibly some better separation is funished by boiling as Iron floats to top with a froth & that filters clearly

Took Residue & washed it then filter & boiled it 20 min refiltered & washed several times hot water till no line about 300 cc water this only shows lime faint

Will treat residue with HCl — in excess after boiling 1/2 hour get faint lime like 62 mg standard, about 250 cc water + HCl — Spod 21 is little stronger than analysis No 22

No 21

Quenched Spodumene, then ground fairly fine & mixed as follows, fused 1 hour at yellow white. —

25 grms Spodumene
25 Salt
37 gypsum
87 — Quenched water & whole soap to dry & powdered very fine —

Weight 76 grams lost 11 grms or 12.67 % lost

Spod dont filter as fast as Analysis of 22

Dont blue red litmus or redden blue litmus

Final Residue treated strong acid all night, Lumed diluted to 800 cc to equal 20 mlgm standard CaOH 250cc —

OVER

7000 cc Equals 20 Mlq Standard
Equals 112 lbs di OH Tan
[illegible]
Yield 90% by Water
If fired 2 hours thick wood
get 95% or more

It's necessary to vigorously boil for a couple hours long boil any dil Cl for 20 minutes — filtrate clear now some chemical reaction for the better takes place or circulation is more rapid —

Took residue washed it thru filter & boiled it again — for 20 minutes — about 300 cc good lime after washing reaction on filter or immediately after lime till lime disappeared entirely —

I will not treat residue with HCl — in excess don't think much left

after boiling acid ½ hour get a lime faint about like 62 mg standard — about 250 cc liquid or less

22

Ambligonite done same as 21 but

25 ambig
25 Salt
40 Plaster Paris —
90 —

Weight 75 grms lost 15 grms
15.6% loss

fillers better than 21 —
Changes neither Red or blue Litmus —

I diluted the water filtrate with 10000 cc when it was about equal in brightness + drying away time for 2 drops of Pelinson 20 mg LiOH dry in 250 cc acidulated with

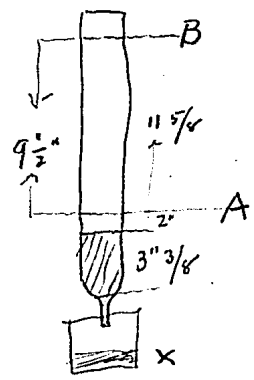
HCl by me, [illegible] Water
Equals [illegible]
Equals [illegible]
I now find it wants more water
have put in 13500 CC
to Equal Standard 20 Meq 250 cc

This is 54 times stronger which
gives 1.080 grms in the 10 grms
of ore, multiplied by 90000 times
gives 97200 grms or 216 pounds
of Li OH dry Extracted from the
ore by water —

The acid Solution Excess HCl all
night + washed well —
1100 CC Equals 20 Meq Standard
This is even higher Extraction than
#21 Spodumene which appears
to be the most difficult + wants
longer fusing probably
1% left in ore or 93% Extracted —

Saturday 5 pm Dec 4th

Put in a charge of OTE 25 25 37



inside diameter one inch.

Cold water

at 9 30 am monday 6th

found water had gone down from B To A - 9 1/2 inches 38 hours -

filtrate strong Li Line, water coming thru now gives no Li Line

Either I made a mistake in 21 & 22 by taking all of the fusion instead of 2½ times less or less yield due to not grinding fine before fusing —

23.

25 Lepidolite. not reground before fusing — probably 100 mesh —

25 Lepidolite
25 NaCl
37 Plaster Paris
all of this used

Weight 75.070 grams loss 12 grms fused to a liquid, 1 hour bright yellow verging on white —

This is to ascertain if it must be ground fine before last fusing —

at 4000 cc Equals 20 mg per 250cc standard for water soluble. This is for whole of solution diluted to 1000 cc + 400 cc used in test to represent 10 grams Lep 16 Tol
64 lbs Lith.

25) 64.00 (2.56
145
120
150

24

25 Lepodolite.
12 Salt
25 Plaster Paris = all of this batch used

Very dry did not fuse but fritt

Weight 54.12

Loss 8 grms abt,

Put all in Crock — cc
Total 4000 cc + percent
this showed only Equal
to standard of 20 mg
or 25.6 lbs Lith to
Ton — Something
wrong —

25

25 Lepidolite.
12 g. Salt
35 g Plaster P
all of this batch used
very dry – Did not fuse
fritted –
Weight 66 grms
Loss. 6 –

Diluted whole fusion to
7000 cc to match
standard 20 mil/250cc
1 to 28.
per ton.
112 lbs Li OH
about 1/2 comes out,
or is lost,

With fresh lime & CaCl all night found strong lime in morning its been acting on it all night #28 is very much poorer hence it may be fluorine absence in Spod makes Spod work with CaCl + CaO no fusion whereas Lepid of 28 has Fl. & fused & gave its Li —

4th — & all put in Crack 3000 cc gives about Equal to 40 Milg Standard 12 to 1 — 96 lbs LiOH. or 3% ore =

This is less than #28 but this is Spodumene — + all that is necessary is finer grinding + floating =

5th following on — only all night boiling dish pipe burnt in morning cold get lime 20 milg standard well heat to boiling — about 200 cc lime = to 40 standard — abandoned Residue

#26

Jany 8 4 pm

Put 10 grms lime ground Spodumene that had been roasted & quenched, 1

Put it in small beaker with 250 cc water & perhaps 5 grams Calcium Chloride & 6 grms about of WVa Carbide & boiled it till 6.50 pm when I put it ~~with~~ on butter dish + CaCl overnight — I noticed the Lithia Line rapidly got stronger & stronger until the End it was much stronger than 40 Milg Standard in 300 cc — This is 1% out of ore it looks that if lime is given most all LiOH will come out — Especially if ground very fine —

930 am — 240 cc of sol. to 10 cc diluted it by adding 40 cc to get line between 20 & 40 milg standards — probably 30 standard 1.4% out, @ 24 lbs LiOH.

27

10 grms Ambliganite
about 10 grms Calcium Chloride
& 6 " Caustic W Va Lime
Boiling — on flame
burner at 1040 am
Jany 9 =

Been on hot plate about 2 days
on boiler cloth — Now 7 Gal
for 1/2 hour — over flame —
2nd filtrate shows very
v faint —
Hence Ambliganite must
be fused with the Ca + CaCl
using raw N G

Perhaps can get more out on 3rd Run

On 3rd Exhaustion I put 5 or 6 grms of CaCl in water before I did so, got a line in 250 cc equal to 20 mlg standard, after boiling 1/2 hour showing it wasn't all out + must be boiled as all night at 150 nig —

Even after 4 to 6 oz I get no line with 250 cc but the float stuff on top which under microscope is about 1 to 2/100 dia + glassy - gives a strong lithia line in the froth, perhaps it should be fused 4 or 5 hours to get these attacked,

4th - cocakes just get line very faint with 1 drop + 2 drops gives it ok

Shall make a 5th = 7.400, CC

Equals 40 Mlg Standard

See next page

28

20 grms Ore.

20 grms Lepidolite
15 " Calcium Chloride
10 " Caustic Wash Lime

Fused 1 Hour Weight after fusing 41.590
no HCl came off!
I exhaust the whole, using 500 cc + boiling over flame stand —
Dec 9 2.30 pm

Nothing comes out on 2nd washing all night that can see put filtrate with 1st filtrate.

Diluted 1st + 2nd filtrates to 4500 cc where it equals 40 Mlg Standard, perhaps a little better — 18 to 1 — figuring on 10 grms only or 1/2 ~~Got~~ .72 lbs Li Oxide per ton

should be for 3% ore 96 lbs

This for 3700 cc 10 grams ore
is 118.3 lbs Li_2O.
if 3% 60 lbs Li_2O or 96 lbs Li_2O.
if 4% 80 lbs Li_2O or 128 lbs Li_2O.
Apparently the Extraction is
good —

29

20 Lepidolite.
27 Cluster Pacus
15 Crystals Ferrous Sulphate
25 Salt —
87 1 Hour fuse

Weight after fusing 63.430

Ferrous Sulphate had 7 H_2O in

1st Filtrate Strong —
added a little Salt to mix for
2nd filtrate. —

250cc only line less than 20 standard
after cooling in morning —
Have abandoned Residue as
not pleasing —

Equals 40 Mlq Std 4000 cc
pear —

I put the 3rd filtrate with 1st & 2nd as there are probably some Li left in it about 250 cc -

I now put 10 grms MgCl in & boil for 3/4 hour -
got a faint line - MgCl apparently working - will boil longer
Started 3.47 pm -
off 5 pm - with wet asbestos get the line faint showing further action, This is 4th filtrate will put this by itself -
& try 5th boil with Chloride Barium -
ChlB in at 5.42 pm boiling
617 pm stopped & filtered
no line when very wet.
Will filter off - & try MgCl again
after 2 hours poor, 2 drops to get a faint line - put filtrate with big lot
& No Residue added 20% NaOH 2 25 pm boil

8000 cc equals about 33 cc
Standard, 32 to 1 84 lbs
Li Oh per ton - less than 3%

30 See next page

25 Lepidolite not ~~fuse~~ healed in muffle but raw ore - ground & through 100 mesh only then fused
37 Plaster Paris
25 Salt -
87
Weight after fusing 69.960

1st filtrate strong line
2nd filtrate, weak -
both together very strong
3rd after 1/2 hour with added MgCl
no line - I then put in a little CaCl & boil -
boiled 1/2 hour no line yet ~~froth~~ gives powerful line for long time
I will try other things on this residue ~

C913

This is promising ~~+ ore~~
ground 1000 float mesh think
it will be OK — if MgCl be
used, to leach

Still have residue of 30
Running —

Later — I acidified the 8000 cc
& found it equals to 40 cc Eluant and

or 102 lbs Li OH per ton
or over 3%

Residue has NaOH in it
now — 4 pm —

Nothing came out.

~~I have put strong HCl on Residue~~

Now strong HCl — to see if
any Li — after 2 hours
250 cc line probably equals
60 mlq — Abandoned

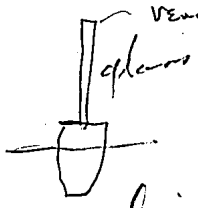
31

20 Lepidolite.
25 Green Ferrous Sulphate
2 Charcoal

1 Hour fusing

Residue weighed 24.320

very near to Lithium



poor line — I will now add CaCl Calcium —

about 5 grms more or less — to see if better — boiled 15 min strong line — about 200 cc filtered + put 4 or 5 grms CaCl in mix for 2nd round —
11th 5 pm for Sunday

last week got faint line
20 mlg Standard 200 cc then
beaker broke & lost 1st part
that run then. - saved that
from 2nd run then but must
be poor — put it with all
the least — The float
in filter gives strong
Lithia line —

3750 CC Same as 40 Mlg

120 lbs

nearly 4%

& I lost some by
cracked beaker
on heater & also by filtrate
beaker —
just for fun put precipitate back
on hot plate for the night —
after 15 hours more get a 30 line with 850 cc but
mud gives much more brilliant line than 33 where
$MgCl_2$ used — My Conclusions in that line

next page

10 grms muffled Epsomite
not fused in little Crucible —
10 grms Lime Caustic
W Va — Nothing Else —

250 cc 1st filtrate strong line
& stays long time probably
$LiOH$ + not Chloride —
This is strange —

Sunday night sometime
bottom beaker cracked & all
sol lost but most of ores
in — put fresh water
on 9 30 13 June
June 15 on all day + night —
2nd filtrate — line better than 40
I put it back on plate for 3rd filtrate

must have $MgCl_2$ or other Chloride to harden the leaching all the time will do it with Lime alone on spot —

Note

I am pulling the mud back & use some $MgCl_2$ to see what next soak does —

4000 cc, but not so strong as 40
3500 is about 40 standard

Last leach taken off 4 pm 17th find better line than 40 with 200 cc — 500 cc not so good —

Think 3750 cc is Equal to 40 — mud well and HCl faint with ACl still pretty bright shows not all out —

Abandoned

to Lott for assay

Weight of Mud Lime & ore
20.5255 grms —

Ott took 1 grm assays
chocos 0.858% L_2O slice
left in ore

This is four as ore was not floated

LWS

I put in about 10 grms what I suppose is chl Mg from made Evap desh on L Otto Table, it gave strong line of Ca —; ? Is OK to put on all night direct on hot plate

200 cc stronger than 40,
15 Dec 5 pm
~~with~~ put residue back for another with more MgCl —

~~[illegible]~~ at 2 pm next day 16th 300 cc found very strong line, been dried on hot plate - glass was lost only little H_2O by evap —

Shows must boil with reflect Condenser for several days or when very fine float 1000 mesh say Hot 3 or 4 days when I think all will come out,

See page 00

Not fused

33 Jany 13 -

10 grams Spodumene —

Not fused in small Crucible

10 grms Canada Lime —

Set aside 11th Jany 5 pm to work at ordinary time being most sol in Cold water —

15 hours cold 250 cc barely get line, evidently requires heat
Jany 13 9.50 am put on hot plate direct —

((by mistake I put 10 grms MgCl in it — after half 4 hours

Jany 14 3 pm - 250 cc very strong line -
Again put more MgCl - get line again " MgCl get a line — ~~will put it away (Results) for night as have no room~~
~~MgCl~~ —

I put it back on hot plate with 2 grams of $MgCl_2$ for further soak as the float mud still gives good lime —
on 3 30 pm 16th —
17th 9 30 am - about 200 cc lime
about 30 - float stuff gives strong lime -
I suppose it will come out slowly but too much time would be required
I will now determine the Total cc. of all washings

NOTE = The residue is now Slimy - very slow to filter
will have trouble here in factory process —

4000 cc is just as good as 40 standard -
16 to 1 640 mlg LiOH in it
128 lbs - Exactly 4% Li_2O in ore
got out
This is better than 32,+ is due to $MgCl_2$
but is not very much better,

I save the slimy residue
dry it & see what can be done to get more out as slime shows strong lime right in flame

Continued - OH assayed the mud residue -
Weight of total residue 21.76 grams
LiOH using 1 gram of the mud. got. 0.835% Li_2O.
in ore,
This is not floated but was fused in small crucible —

(4.)

Not found

34

10 grms Opodeumene

About 15 grams Sulphate Magnesia

Jay 14 3 pm frothy up

200 cc not equal to 40 — abandoned

36 - not found

10 specimens
about 15 grams Mercuric
Chloride -

Jany 14 3 pm -
Dark all thru liquid
ng - 150 cc filtrate only
get 40 lines - abandoned

1st filtrate 5 pm 16th 350 cc very strong lime
+ further washing quite strong lime —
100 more cc — then weak.
Now on 2nd leach with 2 or 3 grms MgCl —
decant on plate 250 cc water —
5 20 pm —
off. 10 am — filtrate 300 cc about 40 lime
mud shows dull lime — dilut by with HCl —

Put residue back with 1 grms $MgCl_2$
+ balance of Lime
at 11 am 17th — for 3rd leach —
but think there is very little left —
I think 50% comes out with less
than 1/2 the $MgCl_2$ + Lime
I leave 3rd leach on all
night, beaker 1" of Top
3rd filtrate, 250 cc = to 20 standard

All together 3000 cc about
Equals 40 standard — of anything
is weaker than 4° mud with HCl —
not very bright, think got 90%
off all in it + LOH was among
final residue —
Think it may have reverted
as it was 24 hours cold

37

LOH — Concentrated residue after
fusing + exhausting gives about
10 cc with poor lime — 99%,
This is perfect exhaustion

10 grms floated Lepidolite

White poor quality, brot to yellow in
muffle, quenched in water, ground +
floated — till all about 1 to 1½
thousandths in diameter

Total Lime to be used W Va Carbide
10 grams only a little at a time
used, Also 3 grms $MgCl_2$
used only little at time used,
about 100 cc water maximum

On 3.15 PM — 15th ½ grm MgCl 1 grm
CaO —
at 6 pm put in 3/4 of the Lime
+ 2/3rds of the $MgCl_2$ + increased
water to 200 cc set on
boiler plate for all night —
at 2 pm 16th took off boiler decant put decant
on plate, 4 pm added a little $MgCl_2$ + lime —

2nd filtrate Couldn't get lime Except
Extra drop on asbestos —

3000 cc about same as 40.
may be a little weaker

This is best
of LOTL fusion of
residue & Smith
Method, first filtrate
10 c only dull lime
shows 99% Extraction
of Li by this process

38 LOTL Concentrated filtrate
from fusing residues —
10 cc lime dull — ~~[illegible]~~
~~[illegible]~~ perfect Extraction 99%
Lepidolite floated Exactly as 3

10 grams Lepidolite
10 grams Lime
5 grams Chloride Calcium

The lime ½ & CaCl ½ put in with
ore for 1st soak — 250 cc water
Jany 16 6 pm on hot plate direct.

1st filtrate 11 am 17th —
put residue back with
some CaCl + lime for 2nd
leach —

Mud gives weak lime after washing
well — with HCl on mud gives fairly
strong lime — showing not all
attacked —

I think 80% comes out with lean
liquor ½ the lime & $CaCl_2$ —
2nd leach on 1150 am — stays on all
night till 18th beaker 1½" of Tops

CRT

as 40 standard appears better a little than #37 —

39

10 Lepidolite like 37 —

10 Lime —

10 NaCl —

250 cc H_2O

only about 1/2 of lime & salt put in on 6 20 pm dec 16 on hot plate Dec 16

1st filtrate off 1130 am —

Mud without HCl gives no lime — with HCl — strong lime, showing all not acted on —

I think 80% comes out with less than 1/2 the lime & salt

2nd lead on at 12 noon — I leave it on all night till 18th —

3rd filtrate, 300 cc concentrate get lime — put all filtrates together it appears 3000 cc is as good

CIRC

Note =

37 38 + 39

appear to all have the same strength should say each was close to 3000 cc with a line a little less than 40 mlg dry LiOH in 250 cc standard

2750 cc would be certain = In all cases I acidulated the filtrate with HCl before testing in spectrum

It is possible that neither $MgCl_2$ $CaCl_2$ or NaCl is required to get the same result, with Lime Caustic alone + the product will be the Lithia hydroxide Caustic

2nd Leach I put in 2½ grams more of lime being ½ or 5th gram so far —
3rd leach put in all the lime —
on 3 hours 250cc lime better than 40 — This shows must have Enough lime at first to fuse Combine with all the SiO_2 Al_2O_3 + Fluorids + some Excess
Now on for 4th leach —
all night direct on plate —
300 cc —
4th 250cc lime about 30 —
filters slower than usual but may be due to fact I allowed it to Cool down to determine settling point

Phenomenon = An Electrolyte with Solution seems to hasten the Exhaustion. See 37-8+39 —
probably only a little is required = Later Chlorides increase Solubility of lime
Now on for 5th soak — get lime
All put together 3000 lime
not Equal to 40 standard should say 30 only — showing not all acid must have Chlorides for rapidity

40 — There is no doubt we must have stirrers in tanks but they can go slow To prevent incrusting bottom + also the keep each particle in saturated lime solution

10 grams Lepidolite like that used in 37-8-39 — fluxed 1000 @ 1500 mesh not fused in little Crucible but ignited + quenched
10 grms Lepidolite
10 grms Lime if necessary
Nothing Else —
On hot plate 6 pm 18th
250 cc water + about 2½ gram of lime to start it —
1st filtrate 10 AM 350 CC after HCl. good lime —
Noticed large amount hard at bottom in practice must be stirred by plows

No 40 with 10 grms Lep
+10 Lime - 4th Extraction
after taking off was allowed
to settle down, in 15 min
it settled in beaker so white
float occupied 6 sqr inches
This is about 312 Cubic
feet per ton of ore & ton of
Lime -

The bulk of ore (float) for 10 grms
Dry occupies about 1 inch
the unslaked dry Caustic Lime
about 1 inch 3/16 inch -
+ the mud of the Leach
will probably settle
to 5 @ 6 inches - (ie) the
dry ingredients finally
settle to 3 times their original
Dry bulk

41 -

10 Lepidolite
10 Lime
1¼ inch — Water — in beaker, lime slaked

Cold Leach

on 2.30 pm Jany 22 —

First water used 1¼" in beaker was hot Lep 10 Lime 10 all together then water then slaked Lime — Now allowed get Cold & Continues —

Tested Feby 9th 200 cc
Not equal to 40 —
Cold dont work —
If heated hot under say 50 lbs it would go much faster Heated at 212° not Hard

Σ

(1335)

10 Lep
10 Lime
Minnamam water to slake lime
Put on plate on butter dish –
Jan 22 2.30 pm –

10 Amblygonite Floated —
10 Lime
Beaker full water for night run

5 30 pm — Jany 22

1st filtrate of 1110 cc — ~~follows~~
~~water above clear 1 3 4 5~~ —

2nd filtrate —

No lime at 2000 cc

NG —

Lime itself NG on
dmbleq

I put back residue
& put 5 grams Barium
Hydroxide in it at
4 30 pm
2 hours no line visible
abandoned -

43 -

1000 mesh

10 grams Ambliogonite (floated)
10 " Lime
2 ½ grams Calcium Chloride

Full beaker for all night Leach
5 30 pm - Jany 22 1919

1st Leach - filters fairly well -
not so fast as Spod or Lep but
good. its finer & more gelatinous
off 10 am Jany 23rd
on for 2nd at 1150 am
23rd off 10 am 24th
all filtrates - 2000 cc equals
= 40 NG -

CaCl shows better than
Salt or MgCl for
curbing & probably
on Spod & Lep

10 grams Ancleligsuite floated
10 grams Lime
2½ grams Salt NaCl
Beaker full water for all
night leach

5 30 pm — Jany 22 1919

1st filtrate - filters very well
not so fast as 40 or 45 p it
fewer + more gelatinous
off at 10 am 23rd

On for 2nd at 1050 am 23

2000 cc weaker than 40
probably 30 NG

45

10 grms ambligonite floated
10 " Lime

2½ grms Magnesium Chloride

Breaks full water for
all night bleach —

5 30 pm — June 22 1919

1st filtrate off at 1115 am +
filters slower than 42-3-4

2nd at all 2000 cc

~~Sto~~ Very faint Lime MG

46 -

Spodumene floated
1000 mesh

10 Spodumene
10 Lime
1 gram Cane Sugar
On at 2 30 p.m. - July 23 -
1st pass line - Good work

(157)

47

10 Iodum, floated
10 Lime old WV not lively
1 gram glycerine —

This has a fairly strong line, ~~[illegible]~~ lasts to 3 times stronger than 46 & may be something in it — it was semi caked together so whole balloon moved about inch high in beaker — weight very hot all night as I turned steam down a little & set beaker on slope

out 10 am —
all filtered - 2500 CC
same as 40 mlg
There is more in milk
not well exhausted too cold
all night —

Shalers Lithia Water all
night with Lime but always
no sign of being attacked—

48-

10 grms anthracite - floated 1000 mesh
10 " Barium hydroxide -
2 ½ grms salt,

On in 250 cc Jan 24 - 3 pm -

This is not equal to
2000 cc 40 slum
Run passed -

C1017

49 =

Note white float dont seem to settle down in 4 hours it might in 24 —

Put it back on hot plate for hour then filtered it & washed till got no lime —

Diluted to 8000 cc the line equals 40 mlg standard & probably a little better —

I put residue with 5 cc more HCl, back on hot plate to see if more comes out, Saturday 6 pm 25th

3 pm 26th = 250 cc line not so good as 40. so practically got Lithia out on the other breakman's

What's needed is fine float & agitation by stirrer while leaching — possibly grinding on bottom, or a separate

most important if open final results

49 — OK Process

10 ounces floated
15 cc HCl
2½ grams oact
250 cc Water

300 cc strong line — there is lot white float makes it filter poor not 1st drop by drop — only filtered a little put it back for the night 6 30 pm 24th = Looks as if stirrer was necessary otherwise each particle coats with an impermeable coat & stops action —

took out residue & ground it in agate mortar then put back on all the filtrate on hot plate

Now determine
Minimum HCl — dilute 5 cc + H2SO4

50 –

10 Anthraquinone floated
10 gms Strontium Hydroxide
2 1/2 gms Salt –
250 cc Water
on July 24th –

filtered 1/2 goes thru clear
shows pretty good line
3/4 hour on heater –
On 6 pm 24 for all night

Even after grinding
in Mortar only 2000 cc
as good as (40 mlg standard)

[165]

51 = will put the 2nd leach in when ready & determine —
Hole got in beaker lost some say 15% of residue
all together —

7000 cc = 40 —
Think H_2SO_4 as good as HCl — & lost some
H_2SO_4 will work OK

51 =

10 Amblygonite floated
15 cc Conc H_2SO_4
250 cc water —
on 26th 4 30 pm —

off at 10 am 27 = filtered & ground residue — put on hot plate 11 am 27 — with 5 cc more H_2SO_4 Conc was not much residue & that was fine — had fine white float acts same as HCl — Diluted to 3000 cc strong line 27 sec — had use spat to bring out lem

52

The mineral precipitate on particles does not break away to allow fresh action when stirred this way — it does better than still — would in time do the treatment

I am now making a leacher like the old washer pan with 4 shoes rotated grinding on the bottom of the pan — I feel sure this will solve the problem —

52

10 amblig floated

10 W Va Lime —

½ put in at 1st —

beaker has 3" water.

on hot plate with rotating paddles to keep it agitated & grind off reaction film

On at 210 pm = 27 m —

[illegible] = 40

[illegible] of [illegible] to [illegible] = 4250 4

OR

2000 cc lime not equal to 40 mlq standard —

53.

10 grms Felspar (Trenton) Floated
10 " WV Lime Caustic -
200 cc aq Hot plate at
9.50 am - 28 Jan

~~10h [illegible] to [illegible] at~~
~~Chef or stock [illegible]~~
~~[illegible] to bottom = [illegible]~~
~~[illegible] broke [illegible]~~
~~lost~~

(170)

54

10 ambliq floated
5 grams 66° H_2SO_4
200 cc aq
11 15 am — 28th
acid too weak
burst beaker by bumping
lost

55

10 amblygonite flooded
7½ grms 66° H_2SO_4
200 cc aqr
1115 am Jany 28

Acid was too weak
bumped in night &
lost.

56 =

250 cc no line ~~of~~ 2nd filtrate

I then used 1st filtrate

+ had to dilute it to

9000 cc ! & got

line equal to 40 Vol Standard

This shows Ton of ammoniac

will be exhausted by a

Ton of H_2SO_4 — I now must

see how much less will do

it — ! I acidulated the

9000 cc filtrate with some HCl.

dont know if it was necessary

Phenomenon

Ok process

56

10 ammoniacal —

10 grms 66° H_2SO_4

200 cc aq —

1115 am 28th

Acid was strong enough

to prevent bumping

+ it was OK at 830 am

29th —

Notice that filtrate runs

clear even on start of filtering

Strong line 300 cc — Residue

I put 15 cc 66 H_2SO_4 —

Equals 9 grams by weight I

think as there is considerable

residue — back on hot plate

915 am —

10 grms Lepidotile fluated
10 grms Chalk ——
$2\frac{1}{2}$ grms about $CaCl_2$

only little comes out, 300 cc = 40

58 =

floated

10 Ambliqenite
15 Chalk
5 Chloride Calcium

Only little Coma
out, 500 cc = 40

C1817

59

About 10@15 grains
Glucoside not powdered
the clean green
10 Lines -

60 -

10 amblygonite floated
10 Lime
2 ½ Calcum Chloride

In grinding Pan leaches
2 15 pm - Jany 30 1919
Stopped for the night heat
off - started again 31st -
off 6 pm - filtered -
about 2 pm I added 5 grms
more of slaked lime -
fearing 10 grms might not be
enough -
It filters OK - ~~Residue~~
dark from iron I suppose
Total Water was about 400 cc
only 2000 cc Failure
Perhaps must have big weights
these small ones destroy group -

(115)

61

10 Ambliqonite floated

S: 20 Lime.

5 $CaCl_2$

S: 850 CC water

on 9.30 am 31 January 1919

to see if action will go on further than 1/3rd as heretofore,

" at 10 10-qot lime = 20 kilg-

Very poor froth 10 cm —

Put in 15 grams Salt-

62 –

fine ground Glucoride, green,
not floated –

9.360 grms –
10 grms Lime –
2½ Salt –
250 H_2O –

on 230 pm – 1st 7s6y

It only takes out very
little, – 1/8 of what
it should must be
Combined with the
Iron somehow

C109